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(A) Non-woven activated carbon fabric.

(5) An air and water vapor permeable, toxic vapor absorptive non-woven fabric material comprising a wet-laid sheet containing fibrillated acrylic fiber, and an activated carbon constituent selected from the group consisting of activated carbon fiber and activated carbon particles.

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NON-WOVEN ACTIVATED CARBON FABRIC BACKGROUND OF THE INVENTION

The need for protective clothing has long been recognized by both military and commercial personnel. manufacture and use of certain dangerous chemicals such as pesticides, toxic materials etc. in the form of sprays or mists, gases etc. requires that personnel involved therewith be provided the safest protective clothing available. Protective clothing has, in the past, been manufactured from completely impermeable materials such as plastics or oilskin. These products did not prove sufficient however, due to preventing the egress of heated air and perspiration from the wearer. Other attempts to provide protective clothing involved the use of absorbent charcoal or other materials in particulate form, however, these products also involved difficulties due to the loss of the particulate material over a period of time. use of quilted woven carbon cloth and adhesives to bind the carbon particles, also was less than commercially successful, see U.S. Pat. Nos. 3,586,596; 3,769,144.

Accordingly, if a fabric configuration could be developed which overcame the disadvantages of previous fabrics, a step forward in the art would be achieved.

SUMMARY OF THE INVENTION

Non-woven fabric material containing, as the toxic vapor absorptive ingredient therein, activated carbon particles and activated carbon fiber is disclosed in U.S. Application Serial No. 531,366, filed September 12, 1983.

According to the present invention, a non-woven fabric material containing, as the toxic vapor absorptive ingredient therein, activated carbon particles or activated

carbon fiber, is produced via the wet-laying process, utilizing fibrillated acrylic fibers as the binder material, the product being air and water vapor permeable and providing the user protection from dangerous chemicals in liquid or gaseous form.

DESCRIPTION OF THE INVENTION INCLUDING PREFERRED EMBODIMENTS

In accordance with the present invention there is provided an air and water vapor permeable, toxic vapor absorptive, non-woven fabric material comprising

(a) fibrillated acrylic fibers, and

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(b) an activated carbon constituent selected from the group consisting of activated carbon fiber and activated carbon particles.

The novel products of the present invention are prepared by wet-laying the activated carbon constituent (activated carbon fibers or activated carbon particles) and fibrillated acrylic fibers from a water suspension thereof.

In the embodiment of the invention wherein the activated carbon constituent is activated carbon fiber, the concentration of fibrillated acrylic fiber may suitably range from about 5 to about 60% by weight, based on the total weight of the acrylic fibers and carbon fiber, and the concentration of the carbon fiber will be in the range of from about 40% to about 95%, by weight, on the same basis, the total weight of the two components being 100%.

In the embodiment of the invention wherein the activated carbon constituent is activated carbon particles, the concentration of fibrillated acrylic fiber suitably may be in the range of from about 35% to about 65%, by weight, based on the total weight of acrylic fibers and carbon particles, and the concentration of activated carbon particles suitably is in the range of from about 35% to about 65%, on the same basis, the total weight of the two components being 100%.

The activated carbon constituent (activated carbon particles or activated carbon fiber) and fibrillated acrylic fiber are wet-laid using the conventional paper-making process well known in the art. Flocculating agents and surface active agents can be incorporated into the water suspension in order to facilitate the paper making procedure as is also known in the art. The acrylic fiber may be produced from any known acrylic polymers such as polyacrylonitrile, copolymers of acrylonitrile and other copolymerizable monomers such as methyl methacrylate, ethyl acrylate etc; homopolymers and copolymers of other fiber forming monoethylenically unsaturated monomers such as vinyl acetate, vinyl chloride, styrene, vinyl pyridine, acrylic esters, acrylamide and the like.

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Fibrillation of the acrylic fibers, which should range from about 1mm to about 25mm in length, is also accomplished as is known in the art i.e. such as by high speed shearing of the fibers.

The activated carbon fibers are also well-known in the art as are methods for their production. They can be used in lengths of from about 0.3 to about 15.0mm, preferably from about 0.5 to about 10.0mm, and can be prepared from such carbon fiber precursors as coal tar pitch, petroleum pitch, coal tar, petroleum derived thermal tar, ethylene tars, high-boiling coal tar distillates, ethylene tar distillates, gas oils or polynuclear aromatics. Also useful as precursors are polymers such as acrylonitrile homopolymers and copolymers, polyvinylalcohol, and natural and regenerated cellulose. Methods for preparing activated carbon fibers useful herein are disclosed in U.S. Patent Nos. 4,069,297 and 4,285,831, which patents are hereby incorporated herein by reference.

The activated carbon powder or particles, have a particle size ranging from about $0.1 \mu m$ to about $100 \mu m$, preferably from about $1.0 \mu m$ to about $80 \mu m$ and are also prepared from any of the carbon precursors described above.

Regardless of whether activated carbon fiber or ac-

tivated carbon particles are utilized as the activated carbon constituent of the non-woven activated carbon fabric of this invention, the surface area of the carbon constituent, as measured by BET determination, desirably is greater than about 500M²/gm and preferably greater than about 1,000M²/gm. Although most commercially available activated carbon materials do not have surface areas greater than about 2600M²/gm, as measured by BET determination, the so-called very high surface area activated carbons which have surface area values as high as 3500M²/gm are particularly advantageous in the practice of the present invention.

As associated with the desirability of providing a high degree of structural integrity to the non-woven fabric material of the present invention, it is preferred in practice that the particle size distribution of activated carbon particles utilized in the invention should include no more than 15% by weight of particles above 500 microns diameter and no more than 15% by weight of such particles below 10 microns diameter. Correspondingly, the particle size distribution of activated carbon fiber preferably includes no more than about 15% by weight of such particles of fiber diameter below about 1 micron and no more than about 15% by weight of such fibers with diameter above 100 microns. In general, it is satisfactory if the length of the carbon fibers is in the range of from about 100 microns up to about 2 centimeters.

The wet-lay sheet making process (papermaking) used herein for the production of the novel fabric material of the present invention, results in a product having unique sorptive characteristics, a thickness of at least about 0.005, preferably at least 0.01, inch, a high sorptive capacity to weight ratio and high porosity to fluid flow.

The surface of the novel fabric material of the present invention may be embossed during or after its production to improve sheet flexibility and/or porosity. The novel non-woven fabric material may be laminated to a

woven, non-woven, knitted etc. backing such as matts, felts, papers, etc. produced from cotton, hemp, flax, ramie, jute, silk, wool, leather, flannel, flannelette, swansdown, ooplin, cellulose ethers or esters, nylon, rayon, acetates, polythene, glass, rock wool, asbestos, in order to strengthen the material.

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Lamination of the novel products hereof to the abovementioned backing materials may be achieved by the use of water vapor and air permeable adhesives, preferably those available in the form of foams, such as rubber or acrylic latexes, polyurethanes and the like. These adhesives are self-adhering and upon curing foam and set into strong bonds.

The surface of the novel fabric material claimed herein may be rendered hydrophobic by coating with a porous silicone film or a polymer such as polytetrafluoroethylene. Additionally, a reactive coating capable of decomposing toxic agents, e.g. a coating of a sulfonated polymer to hydrolyze nerve gas, may be applied thereto so that the activated carbon particles and/or fibers form a second line of defense.

The fabric material of the present invention has a wide variety of uses. It is useful for protective purposes and for filtration and separation of gases. The uses include the manufacture of the fabric material into wearing apparel e.g., military uniforms; blankets, sleeping bags, bedding, surgical dressings, wrappers and containers, covers, tarpaulins, tents, curtains, gas masks, paint spraying masks, air-conditioning duct filters, flue gas deodorizers and the like.

The following examples are set forth for purposes of illustration only and are not to be construed as limitations on the present invention except as set forth in the appended claims. All parts and percentages are by weight unless otherwise specified.

Example 1

A mixture of 45% fibrillated acrylic fiber and 55%



- 6 -

activated carbon powder in 18 liters of water is formed into a sheet using a standard hand papermaking machine. The sheet is dried under pressure at 70°C to 120°C. The resultant fabric material is then tested. The results are set forth below in Table I.

Example 2

The procedure of Example 1 is again followed, except that 6.3% fibrillated acrylic fiber and 93.7% activated carbon fiber are employed and the paper material is embossed after forming but before drying. The results are set forth in Table I, below.

Example 3

The procedure of Example 1 is again followed except that 19.4% fibrillated acrylic fiber, 80% activated carbon fiber and 0.6% polytetrafluoroethylene are employed. No activated carbon powder is present. The results are set forth in Table I, below.

Example 4

The procedure of Example 2 is employed except that 6.3% of fibrillated acrylic fibers and 93.7% of activated carbon fibers are employed. Two layers of the resultant fabric material are laminated to a 65/35 polycotton fabric utilizing a commercially available acrylic foam adhesive. The results are set forth in Table I, below.

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TABLE I
Example No.

		-				
Property _	S	11	. 2	3	4	
Weight (oz./yd ²)	7.9	12.1	5.0	3.0	9.9	
Thickness (Inch)	0.090	0.040	0.038	0.023	0.064	
Air Permeability (ft ³ /min/ft ²)	46	0.6	.39	21.1	93	
Water Vapor Transmission (gm/hr/M ²)	74	-	-	-	-	
Tensile Test (lbs/in)	36	44	2	6	2	
Flexibility (Taber-g/cm)	3.1	36.5	9.8	14.0	. 22.0	
Static CCl ₄ Absorption (mg/cm ²)	14.9	15.1	5.6	2.2	10.5	١
Static CCl ₄ Sorptive Capacity (% fabric wt.)	53.6	36.9	31.9	21.9	30.1	
Dynamic CCl ₄ Absorption (mg/cm ²)	2.5*	6.7*	0.85	* 0.05*	1.35*	
Dynamic CCl ₄ Sorptive Cap. (% fabric wt.)	9.0*	16.3*	4.8.*	0.5*	3.9*	

^{*} Calculated; based on 0.5 mg/l leakage point

S = Standard commercial product (nylon knit backed polyurethane foam cont. activated carbon powder)

WE CLAIM:

- 1. An air and water vapor permeable, toxic vapor absorptive, non-woven fabric material comprising a wet-laid sheet containing fibrillated acrylic fiber, and an activated carbon constituent selected from the group consisting of activated carbon fiber and activated carbon particles, wherein the concentration of fibrillated accrylic fiber ranges from about 5-65% by weight based on the total weight of fibrillated acrylic fiber and activated carbon constituent, and the concentration of said activated carbon constituent ranges from about 35-95%, by weight, same basis.
- 2. The fabric material of Claim 1 wherein the fabric material surface is embossed.
- 3. The fabric material of Claim 1 wherein a backing member is adhered thereto.
- 4. The fabric material of Claim 2 wherein a backing member is adhered thereto.
- 5. The fabric material of Claim 1 wherein the surface area of the activated carbon constituent, as measured by BET determination, is greater than about $500M^2/gm$.
- 6. The fabric material of Claim 1, wherein the activated carbon constituent is activated carbon particles, wherein the concentration of fibrillated acrylic fiber ranges from about 35-65%, by weight, based on the total weight of fibrillated acrylic fiber and activated carbon particles, and the concentration of activated carbon particles ranges from about 35-65%, by weight, same basis.
- 7. The fabric material of Claim 1, wherein the activated carbon constituent is activated carbon fiber, wherein the concentration of fibrillated acrylic fiber ranges from about 5-60% by weight, based on the total weight of fibrillated acrylic fiber and activated carbon fiber, and the concentration of activated carbon fiber ranges from about 40-95%, by weight, same basis.
- 8. The fabric material of Claim 6, wherein the particle size distribution of activated carbon particles

contains no more than about 15%, by weight based on weight of the particles, of particles with a diameter above about 500 microns and no more than about 15% by weight, same basis, of particles with a diameter below about 10 microns.

- 9. The fabric material of Claim 7, wherein the distribution of fibers having a diameter below about 1 micron does not exceed about 15% by weight of the total activated carbon fiber content, and the distribution of activated carbon fibers having a diameter above about 100 microns does not exceed about 15% by weight, same basis.
- 10. The fabric material of Claim 1 wherein the surface area of the activated carbon constituent, as measured by BET determination, is greater than about $1.000 M^2/gm$.



Application number

EP 86 10 4462

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EUROPEAN PATENT APPLICATION

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6 Filter paper.

A toxic vapour absorptive nonwoven filter material comprising a wet-laid sheet containing fibrillated binder fibre, active carbon in fibre or particle form and submicron diameter glass fibre. The surface of the filter material may by treated with a hydrophobic or an oleophobic agent.

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FILTER PAPER

Background of the Invention

The need for highly efficient filter materials has long been recognized by both military and commercial personnel. The manufacture and use of certain dangerous chemicals such as pesticides, toxic materials etc., in the form of sprays or mists, gases etc., requires that personnel involved therewith be provided the safest protection available. Protection, e.g., in the form of gas masks, has, in the past, not been completely satisfactory in filtering both aerosols and particulates at a low pressure drop. Furthermore, most previously used filter materials do not provide toxic vapor absorption as well as highly efficient mechanical filtration.

Accordingly, if a filter material could be developed which overcame the disadvantages of previous filters, a step forward in the art would be achieved.

Summary of the Invention

According to the present invention, a non-woven filter material containing active carbon and submicron size glass fibers is produced via the wet-laying process, utilizing fibrillated fibers as the binder material, the filter providing excellent removal of aerosols, particulates and toxic vapors. The filters of the present invention have exhibited filtration index numbers over nine whereas previously used filters have exhibited values of slightly more than four.

Description of the Invention Including Preferred Embodiments

In accordance with the present invention, there is provided a high efficiency, toxic vapor absorptive, non-woven filter material comprising

- a) active carbon,
- b) submicron size glass fibers and
- c) fibrillated binder fibers.

The novel products of the present invention are prepared by wet-laying the active carbon, preferably in the form of fibers, submicron size glass fibers and fibrillated binder fibers from a water suspension thereof. The suspension should contain from about 2-20%, by weight, based on the total weight of the filter material, preferably from about 5-15%, by weight, of the fibrillated binder fibers, from about 30-80%, by weight, same basis, preferably from about 40-70%, by weight, of the active carbon and from about 10-50%, by weight, same basis, preferably from about 15-40%, by weight, of the submicron size glass fibers, the total weight of the three components being 100%.

The active carbon, submicron size glass fibers, and fibrillated binder fiber are wet-laid using the conventional papermaking process well known in the art. Flocculating agents and surface active agents can be incorporated into the water suspension in order to facilitate the paper making procedure as is also known in the art. The binder fiber may comprise any materials known to be useful for this purpose, such as polyester fiber, polyolefin fiber, etc., however, the preferred fiber comprises acrylic fiber which may be produced from any known acrylic polymers such as polyacrylonitrile, copolymers of acrylonitrile and other copolymerizable monomers such as methyl methacrylate, ethyl acrylate, etc.; homopolymers and copolymers of other fiber forming monoethylenically unsaturated monomers such as vinyl acetate, vinyl chloride, styrene, vinyl pyridine, acrylic esters, acrylamide and the like.

Fibrillation of the binder fibers, which should range from about 1mm to about 25mm in length, is accomplished as is known in the art, i.e., such as by high speed shearing of the fibers.

The active carbon component of the novel filter of the present invention can comprise carbon particles or carbon fibers or mixtures thereof, carbon fibers being preferred. These carbon fibers are well-known in the art as are methods for their production. They can be used in lengths of from about 0.3 to about 15.0mm, preferably from about 0.5 to about 10.0mm, and can be prepared from such carbon fiber precursors as coal tar pitch, petroleum pitch, coal tar, petroleum derived thermal tar, ethylene tars, high-boiling coal tar distillates, ethylene tar distillates, gas oils or polynuclear aromatics. Also useful as precursors are polymers such as acrylonitrile homopolymers and copolymers, polyand regenerated cellulose. vinylalcohol, and natural Methods for preparing activated carbon fibers useful herein are disclosed in U.S. Patent Nos. 4,069,297 and 4,285,831, which patents are hereby incorporated herein by reference.

The activated carbon powders or particles useful herein should have a particle size ranging from about 0.1 m to about 100 m, preferably from about 1.0 m to about 80 m and may also be prepared from any of the carbon precursors described above.

The submicron size glass fibers useful herein are taught in the art and any known method for their production can be used to prepare them. The range in length is from about lum to about 3500 mm, preferably from about 10 mm to about 2000 mm.

The wet-lay sheet making process (papermaking) used herein for the production of the novel filter material of the present invention, results in a filter having unique sorptive characteristics, a thickness of at least about 0.005, preferably at least 0.01 inch, a high sorptive capacity to weight ratio and high porosity to fluid flow. The filter has superior filtering efficiency for aerosols and particulates and achieves this high efficiency at very low pressure drops. The filter has the capacity to remove toxic vapors from a gas stream by absorption and thus combines improved mechanical filtration through pores The active carbon, when present as a fiber, with absorption. tends to lay parallel to the plane of the sheet, and thus produces a long fluid flow path through the sheet which increases the time available to absorb impurities.

The novel filter material of the present invention may be exposed during or after its production to procedures to provide property enhancement. Thus, the surface of the novel filter material claimed herein may be rendered hydrophobic or oleophobic by treatment with such materials as methyltrichlorosilane, a polymer such as polytetrafluoroethylene, or PFOMA (1,1-dihydropentadecafluorooctylmethacrylate. Additionally, a reactive coating capable of decomposing toxic agents, e.g., a coating of sulfonated polymer to hydrolyze nerve gas, may be applied thereto so that the active carbon forms a second line of defense. Moreover, treatment of the filter with known mildew-cides results in the production of mildew resistant materials.

The filter material of the present invention has a wide variety of uses. It is useful for protective purposes, i.e., for the filtration and separation of gases, aerosols and particulates. The uses include the manufacture of the filter material into components for air filtration systems, air filters for military vehicles and stationary facilities, gas mask cannisters and cartridges, gas purification filters, and the like.

The following examples are set forth for the purpose of illustration only and are not to be construed as limitations on the present invention except as set forth in the appended claims. All parts and percentages are by weight unless otherwise

specified.

Example 1

A mixture of 10% fibrillated acrylic fibers, 80% activated carbon fiber and 10% Type 104 glass fiber in 18 1. of water are formed into a 12" x 12" sheet using a standard hand paper making machine. The sheet is dried under pressure at 70°C to 120°C. The resultant filter material is then tested. The results are set forth in Table I, below.

Example 2

The procedure of Example 1 is again followed except that 10% fibrillated acrylic fiber, 65% activated carbon fiber and 25% Type 104 glass fiber are employed. The results of testing are set forth in Table I, below.

Example 3

The procedure of Example 1 is again followed except that 50% fibrillated acrylic fiber and 40% Type 104 glass fiber are employed. The testing results are set forth below in Table I.

Examples 4-6

The filter materials of Examples 1-3 are individually treated with 1,1-dihydropentadecafluorooctylmethylacrylate and tested. The results are set forth in Table II, below.

In Tables I and II, below, thickness is measured on a TMI Model 551M micrometer gage. Tensile strength is evaluated on a Thwing-Albert Electro-hydraulic Tensile Tester Model No. 30LT. Porosity is calculated from measurements made using a Gurley-Hill S-P-S Tester and Dynamic Sorption is calculated based upon a threat concentration of 760 ppm CCl₄ in nitrogen, a downstream limit of 1.0 ppm CCl₄ and a flow density of 2.5 ml/min./cm². According to the equation:

Break Time (min.) x Concentration (mg/l)

Dyanamic Sorption (CCl₄) = -------

4 x Area of Sample (100 cm2)

The Static Sorption is measured by determining the gain in weight of a sample stabilized in saturated CCl₄ vapor at room temperature divided by the starting sample weight.

Both Pressure Drop and Particle Smoke Penetration are measured on a Q127 DOP Filter Testing Penetrometer. This equipment maintains a flow velocity of 850 cm/min. (32 liters per minute through an area of $37.61~\text{cm}^2$). The Penetrometer generates dioctylphthalate (DOP) particles of 0.3 diameter average at test concentrations of 100 mg/l.

The Filtration Index is a measure of the general effectiveness of a filter and takes into account the compromise between pressure drop and resistance to smoke penetration, i.e.,

Filtration Index =
$$\frac{100}{R}$$
 (2 - Log P)

where R = average resistance, millimeters of water P = average particle smoke penetration, % R.

TABLE I

Filter Paper of Example No.	<u>1</u>	2	<u>3</u>
Thickness (inch) Tensile Strength (1bs/in) Porosity (ft ³ /min/ft ²) Dynamic Sorption (mg/cm ² -CC1 ₄) Static Sorption (% wt. pickup)	.012 1.85 111 0.21 25.8 6.00	.011 2.13 42.4 0.11 22.6	.010 2.05 30.7 0.06 16.0 33.0
Pressure Drop (mm H ₂ O) Particle Smoke Penetration (%) Filtration Index	56.0 4.20	1.00	0.10 9.10

TABLE II

Filter Paper of Example No.	4	5	<u>6</u> ·
Tensile Strength (lbs/in)	2.15	2.40	2.43
Porosity (ft3/min/ft2)	111	43.9	29.1
Static Sorption (% wt. pickup)	14.3	12.8	10.6
Pressure Drop (mm H ₂ O)	10.0	25.0	36.0
Particle Smoke Penetration (%)	42.0°	0.90	0.08
Filtration Index	3.80	8.20	8.60

Example 7

The procedure of Example 1 is again followed except that 15% of the activated carbon fibers is replaced by activated carbon particles in powder form. Similar results are achieved.

WE CLAIM:

- 1. A high efficiency, toxic vapor absorptive, non-woven filter material comprising a wet-laid sheet containing fibrillated binder fiber, active carbon and submicron size glass fiber.
- 2. The filter material of Claim 1 wherein the concentration of fibrillated binder fiber ranges from about 2-20%, by weight, based on the total weight of the material, the concentration of active carbon ranges from about 30-80%, by weight, same basis, and the concentration of the submicron size glass fiber ranges from about 10-50%, same basis.
- 3. The filter material of Claim 1 wherein the active carbon is in the form of a fiber.
- 4. The filter material of Claim 1 wherein the active carbon is in the form of particles.
- 5. The filter material of Claim 1 wherein the binder is acrylic fiber.
- 6. The filter material of Claim 1 wherein the surface thereof is treated with a hydrophobic and/or an oleophobic agent.
- 7. The filter material of Claim 6 wherein the agent is 1,1-dihydropentadecafluorooctyl methacrylate.
- 8. The filter material of Claim 1 wherein the active carbon is a blend of fiber and particles.



EP 84 11 0513

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	DOCUMENTS CON	Page 2		
Category	Citation of document w of rele	rith indication, where appropriate, svant passages	Relevant to claim	CLASSIFICATION OF THE APPLICATION (Int. CI.4)
A	GB-A-1 163 894 *Claim 1*	(D.A.G.MARSHALL)	1,2,4	
A	GB-A-1 173 143 OF STATE FOR DE *Claims 1,3,6 13-23*	(THE SECRETARY FENCE) ; page 2, lines	1,4,6	٠.
A	US-A-3 811 933 al.) *Abstract; Colu	(M.W.UFFNER et	6,7	
A	US-A-2 642 416 et al.) *Page 1*	(A.H.AHLBRECHT	6,7	
A	US-A-3 034 925 al.)	(C.G.DE MARCO et	6,7	TECHNICAL FIELDS SEARCHED (Int. Cl.4)
A	US-A-4 289 513 et al.)	(R.D.BROWNHILL		
A	US-A-4 239 516	(M.KLEIN)		
A	US-A-3 039 914	(P.A.REIMAN)		
A	 .US-A-3 034 947	(J.R.CONLISK)		
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!	The present search report has b	een drawn up for all claims		
	THE HAGUE	Date of completion of the search	NESTB	K.Examiner
Y: part doc A: tech O: non	CATEGORY OF CITED DOCU icularly relevant if taken alone icularly relevant if combined w ument of the same category inological background -written disclosure rmediate document	E: earlier pate after the fili ith another D: document of L: document of	nt document, b ing date cited in the applicited for other n	ing the invention ut published on, or lication easons t family, corresponding



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tegory	Citation of document with of releva	n indication, where appropriate, ant passages	Relevant to claim	CLASSIFICATION OF THE APPLICATION (Int. Cl.4)
A	US-A-2 721 139	(H.F.ARLEDTER)		
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	CATEGORY OF CITED DOC	CUMENTS T: theory o	r principle und	erlying the invention t, but published on, or
· · ·	articularly relevant if taken alone	e after the with another D: docume L: docume	i filing date int cited in the E int cited for oth	application er reasons
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